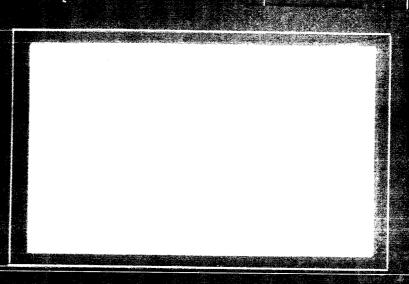
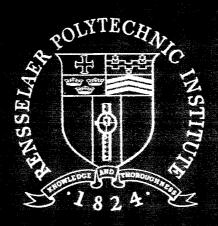
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Rensselaer Polytechnic Institute

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SEMI-ANNUAL PROGRESS REPORT

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

GRANT NO. NaG-100-60

Rensselaer Polytechnic Institute Troy, New York

March 23, 1964

Rensselaer Polytechnic Institute

Semi-Annual Progress Report
National Aeronautics and Space Administration Grant NsG-100-60
September 1, 1963 to February 29, 1964

INTRODUCTION

This sixth semi-annual progress report from the Interdisciplinary Materials Research Program at Rensselaer Polytechnic Institute follows the format established in previous reports. In the ensuing pages each individual research project supported by National Aeronautics and Space Administration Grant No. NsG-100-60, is reported separately. A brief write-up describing the work being conducted and the results obtained thus far is given. No new research programs were initiated during this report period.



RESEARCH PROJECTS

Polymer Research

470.05

Senior Investigators:

Research Staff:

S.S. Sternstein, Ph.D. Assistant Professor of Chemical Engineering D. Hansen, Ph.D. Assistant Professor of Chemical Engineering

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N. Watkins, B.S. Graduate Assistant B. Wasko, B.S. National Defense Education Act Fellow

A. Thermal Properties

Thermal conductivity measurements on a series of polystyrenes of different molecular weights have been completed. These data, and earlier results on polyethylenes have been analyzed with respect to the influences of molecular weight and crystallinity on thermal conductivity of polymers.

One of the research staff, Mr. C.C. Ho, has submitted his Ph.D. dissertation based on this work, and a report is being prepared for publication.

It is planned to do some further work on the effects of molecular weight on thermal conductivity, but the primary effort will be shifted to studies on anisotropic conductivity in polymers oriented by drawing or elastic deformation.

B. Morphology

The techniques developed in this laboratory for thin sectioning and metal staining polymers for electron microscope observations have been applied to studies on drawn and undrawn nylon-66 fibers. These observations have confirmed several facets of spherulite structure in nylon fibers which had previously been presumed from indirect evidence. Based on comparisons between the morphology of drawn and undrawn filaments, a mechanism describing the neck-drawing phenomenon has been postulated. It appears that neck-drawing is quantitatively explained by this mechanism of cooperative crystallographic slip.

A short report on portions of this work has been submitted for publication. One of the research staff, Mr. J.A. Rusnock, has submitted his Ph.D. thesis based on this research, and a comprehensive report is being prepared for publication.

C. High Pressure Infrared Spectroscopy

The infrared spectra of several polymers are being studied at high pressures. A pressure induced diminution of the intensity of the symmetric -CH₂ stretching mode, with little or no change in the intensity of the asymmetric -CH₂ stretching mode, has been observed in nylon 6-6, nylon 6-10, polystyrene, polypropylene, and polyvinyl alcohol. The effect is sufficient to result in nearly complete diminution of the symmetric -CH₂ mode in nylon 6-6 at 25,000 atmospheres. A mechanism has been proposed for this phenomenon and has been submitted for publication together with the data for spectral shifts of hydrogen-bonded polymers as induced by pressure. (This work is partially supported by a grant from the National Science Foundation.)

D. Mechanical Properties

An experimental technique has been developed for studying the growth rates of large macroscopic crazes in various polymers below their glass-transition temperatures. The fracture morphology of these samples is being studied by light microscopy and has shown that a fracture surface, when allowed to propagate through one of these large crazes, shows essentially no ductile flow, but rather almost a pure brittle failure. The theory being developed for fracture of amorphous polymers above and below the glass-transition temperature is being applied to these data, and it appears that the data corraborate the predictions of the theory. Considerable insight into the mechanisms of failure in amorphous polymers has resulted from these studies, and extensions to various modes of failure, and failure criteria, are now in progress. (This work is partially supported by the Institute of Paper Chemistry.)

A generalized formulation for a constitutive equation, for a discontinuous array of structural elements, has been developed from a minimum energy criterion. This theory will find applications where it is desired to predict macroscopic mechanical properties from structural parameters, for example, in the paper-Cellulosic fiber relationships, or in the relationship of macromolecular parameters to the properties of a bulk polymer.

Several experimental studies are in progress to test the theory's validity. The dielectric spectra at low temperatures, and the dynamic mechanical spectra at various frequencies, of copolymers of polyvinyl acetate-alcohol are being studied. Also, the effects of various solvents on the torsional moduli of semicrystalline polymers are being investigated, and the stress-relaxation studies on hydrogen-bonded polymers are being continued. (This work is partially supported by a grant from the National Science Foundation,)

A Study of the Interaction of Dislocation with the Discrete Second-Phase Particles in Dispersion-Strengthened Alloys

Senior Investigator:

G.S. Ansell, Ph.D. Associate Professor of Metallurgical Engineering

Research Staff:

C.J. Barton, B.Met.E. Graduate Assistant
N. Ramchandran, B.S. Graduate Assistant
H.S. Kim, B.S. Graduate Assistant

The objective of this program has been to delineate the strengthening mechanism associated with the presence of a distributed second-phase in a crystalline matrix by both theoretical considerations and experimental observations of the interaction of defects with the

second-phase particles.

During this period, research efforts have centered principally upon experimental observations utilizing transmission electron microscopy. The specific areas investigated include:

- 1) Recovery and Recrystallization Kinetics in Two-Phase Systems Utilizing hot stage transmission electron microscopy and cine and sequence recording techniques, the rate of substructural recovery of cold worked structures and the interaction of migrating grain boundaries with the second-phase have been studied. This work is still in progress.
- 2) Work Hardening Behavior in Precipitation-Hardened Alloys Transmission microscopy studies of the effects of
 matrix-dispersed phase interface characteristics have
 been under investigation on the Al-Zn system. This
 work is still in progress.
- 3) Theoretical Studies Theoretical studies of dispersion-strengthening based upon dislocation theory are progressing on a continuing basis.

Nucleation in Liquid Metals

470.10

Senior Investigator:

W.J. Childs, Ph.D., Professor of Metallurgical Engineering

Research Staff:

J.J. Frawley, M.S., Graduate Assistant S.S. Kale, B.S., Graduate Assistant

The relationship between the vibrational energy required to nucleate supercooled bismuth and the amount of static supercooling obtainable has been studied. Specimens capable of high degrees of static supercooling require more energy to nucleate at a given degree of supercooling showing that nucleation is affected by a combination of the amount of supercooling and the presence of catalysts. The experimental equipment has been modified to further minimize any vibration in a horizontal plane, and also to permit operation in an inert atmosphere.

Bismuth crystals which had grown after nucleation of the supercooled liquid were studied by means of X-ray diffraction. Triplane crystals were shown to grow in the (111) direction with the triplane elements coincident with the (110) planes. The habit of dendrites which grew along the crucible wall were (111) planes with a (211) growth direction.

Ultrasonic Research

470.11

Senior Investigator:

H.B. Huntington, Ph.D. Professor of Physics

Research Staff:

Z.P. Chang, B.S. Research Assistant

The program is planned primarily to investigate the elastic properties of crystals. The principal project is the measurement of third order elastic constants of alkali-halides by observing the influence of uniaxial compression down the (111) axis on the ultrasonic velocities. This direction was chosen because it involved no resolved shear stress on any of the usual slip systems of these structures. We have measured the compresional and both transverse velocities in the (211) and (110) directions but these six results give only five independent results to determine the six third-order elastic constants. We have developed the Cauchy relations to the third order. Of the three resulting expressions, one is used to remove the indeterminancy and the other two afford reasonably satisfactory internal checks.

A secondary project is the measurement of the elastic constants of anthracene and naphthalene. Complete sets for both have been obtained and are now being rechecked.

Ultrasonic Pulse Interferometry

470.12

Senior Investigator:

S. Katz, Ph.D. Associate Professor of Geophysics

Research Staff:

R.S. Gilmore, Graduate Assistant H. Reed, Graduate Assistant G. Randers-Pehrson Student Assistant

The study of the elasticity and density of solids, especially those which undergo polymorphic transitions at high pressures and temperatures, continues to be the principal objective of the present research. The study has been somewhat enlarged in scope.

1) to include those solids which are transparent in thin-section and can be examined optically at high pressures; and 2) to permit simultaneous measurement in electrically conducting materials of elastic wave velocities by ultrasonic methods described in previous reports, and of changes in electrical resistance. A new pressure cell has been designed and built to make possible this simultaneous measurement to pressures of over 100 kilobars. A new 300-ton press has been procured to generate the force required for these pressures.

A small pocket-size press, using diamonds as Bridgman anvils and a stiff helical spring as the force-generating element, has been procured. Calibrating measurements on bismuth, tin, and antimony are under way. It is planned to study the elastic constants and the electrical and optical properties of these and other elements and compounds which undergo polymorphic transitions.

Excited Molecules

470.14

Senior Investigator:

L.G. Bassett, Ph.D. Professor of Analytical Chem.

Research Staff:

R.R. Reeves, Ph.D. Senior Research Associate D.R. Safrany, B.S. Graduate Assistant

A final report on this study has been completed and the conclusions are listed below:

- 1. Metestable $N_2(A^3 \mathbf{Z}_1)$ molecules appear to be inert towards reaction with most molecules.
- 2. Based upon this finding, this species cannot be responsible for certain reactions, claimed by others, to involve this species.
- 3. The reactions of active nitrogen involve, almost entirely, the reactions of N-atoms only.
- 4. Prior to this work, the reactions of hydrocarbons, with N-atoms, were considered to proceed simply via direct formation of a complex, which then rearranged and split off HCN: RH + N→ (RHN)→ HCN + fragments. We have shown, however, that the reactions of hydrocarbons with N-atoms are highly complex, and involve the inter-reactions of several radicals in rapid, highly branching chain reactions. In fact, the reaction of the N-atoms themselves are only secondary, serving only to propagate the reactions of hydrocarbon radicals.
- 5. Prior to this work, the different methods used to determine the concentration of N-atoms in active nitrogen were in disagreement, because of insufficient knowledge of the reaction mechanisms of hydrocarbons. The hydrocarbon titration method was assumed to give only HCN as the nitrogen containing product, and the nitrogen content was taken as equal to that in the active nitrogen.

However, we have shown, also by direct experimental evidence, that HCN is not the only nitrogen containing product formed; N₂ is formed to an extent of over 50% of the N-atoms present.

6. Since the last report was issued, a new effect was discovered. It has been found that the reactions of simple alkanes, which do not react or react only slowly with active nitrogen, are catalyzed by the addition of HCl to the reaction mixture, to such an extent that extremely rapid reactions occur.

Mechanical Properties of Polymers

470.15

Senior Investigators:

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Professor of Analytical
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Associate Professor
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Graduate Assistant
R.J. Boyce, National
Defense Education
Act Fellow
L.H. Vogt, B.A.
Graduate Assistant

A. Rheology

A new study of the flow properties and structure of some low molecular weight liquid polybutadiene and butadiene acrylic acid copolymers has been undertaken. These polymers are being supplied by the B.F. Goodrich Rubber Company and Thiokol Chemical Corporation where they were developed for use in rocket fuels.

The flow properties will be studied over a considerable range of shear rates such that both the lower and upper Newtonian regions can be observed. A cone-plate viscometer is being used for the low shear rate data, 0 to 20,000 sec. -1, while a high pressure capillary viscometer will be used for the region 20,000 to 200,000 sec. -1.

Infrared analysis of the polymers is being carried out to determine their microstructure; that is, the relative amounts of trans 1.4, cis 1.4 and vinyl 1.2 addition. The analysis is accomplished by measuring the extinction coefficients in carbon disulfide solutions for the band peaks at 967, 911 and 724 cm⁻¹ and solution of the equations of Hampton (Anal. Chem. 21, 923 (1949)).

Preliminary results indicate addition occurs mainly as trans 1.4 and vinyl 1.2 with the larger portion being trans.

When the flow data has been obtained, the effects of molecular weight, molecular structure and temperature on the overall flow behavior and energy of activation will be examined in respect to fundamental flow theories.

B. Nature of Water Hydration in Crystals

This study is concerned primarily with the infrared absorption of the coordinated water molecules that are present in hydrated acetate complexes.

A procedure has been determined for the preparation of deuterated hydrates in all cases where a normal hydrate exists. The normal hydrates were dehydrated in an effort to distinguish the absorptions of the coordinated water molecules. The infrared absorption spectra of the anhydrous, dehydrated, hydrated, and deuterated hydrates were compared at room temperature and at liquid nitrogen temperature from 2.5 to 40 microns in an effort to assign all vibrations of the coordinated water molecules.

Free water protons were detected in three of the tetrahydrate acetates while the remaining water protons were found to be important in the formation of hydrogen bonds. Intra- and inter- molecular hydrogen bonds were found to be present in some cases. Low temp-erature infrared analysis showed the broad hydrogen bonded water stretching absorption to actually split into several distinct peaks characteristic of the type of hydrogen bonded water present.

Other than the H₂O stretching and bending absorptions, bands were found in some of the hydrates in the 700 - 900 cm.⁻¹ region which are assigned as rocking, twisting, or wagging modes characteristic of coordinated water molecules. Coordinated water absorptions in the cesium bromide region are assigned to a librational motion of the coordinated water.

Vibrations of the acetate group are found to be fairly standard from complex to complex. The 1300 - 1500 cm. acetate absorption, which occurs as a broad doublet at room temperature, was found to split into the four characteristic acetate frequencies in this region at liquid nitrogen temperature. A series of weak acetate peaks occur in the 250 - 350 cm. segment of the cesium bromide region. The absorptions, which are present in the anhydrous, dehydrated, hydrated, and deuterated hydrates, do not show any sharpening or intensification at liquid nitrogen temperature and, if at all, appear more undefined than at room temperature. These absorptions are tentatively assigned to a librational motion of the whole molecule within the crystal lattice.

The results of this study were presented at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy on March 5, 1964.

C. Study of Ruthenium Complexes Containing Neutral Ligands

In order to determine the nature of the bonding in ruthenium-SO₂ coordination compounds, a complete three dimensional X-ray structure analysis has been undertaken on (Ru^{II}(NH₃)₄ (SO₂) cl)cl. Powder photographs indicate that the analogous bromobromide and pentammine-chloride complexes are isomorphous with the chloro-chloride complex. Intensity data for 1110 reflections have been collected by the counter method using 2 scans. A trial structure, determined from preliminary film data, will be used as a first approximation for a least square refinement program. Bond parameters thus obtained should be accurate to better than 0.01 Å.

Corrolation of the observed infrared bonds with the X-ray structure will indicate the nature of the Ru-S and S-O bonds in these complexes. Specifically, it would be of interest to know whether the Ru-S is sigma bonded or if, as in metal-carbonyl complexes, back donation is significant.

D. Thermal Analysis and Calorimetry of Linear High Polymers

During the past year a fast-heating hot-stage was developed, capable of heating rates of up to 20000 C/minute. These fast-heating rates are necessary to observe melting points of metastable polymer single crystals and dendrites. By heating faster than the relaxation time of the non-equilibrium defects, it is possible to get reproducible melting points which are proportional to the surface and defect content of the crystals. The first material investigated is polyethylene.

With the help of the newly acquired DTA apparatus a second series of measurements have been started, to investigate the change in melting point of polytetrafluorethylene as a function of thermal history. The melting point of virgin PTFE is much higher (20°C) than that of any crystalline material produced by any subsequent crystallization. The preliminary conclusion is that during the initial low temperature polymerization step crystallization to a more perfect crystalline material is induced than can be achieved by later disentanglement of molten chains. It is hoped to present proof of this hypothesis within the next half-year.

High Temperature Alloys

470.18

Senior Investigator:

W.R. Clough, Sc.D., Associate Professor of Metallurgical Eng.

Research Staff:

S.J. Donachie, B.Met.E., Graduate Assistant J.L. Solomon, B.Met.E., Graduate Assistant

L.A. Jackman, B.Met.E., Union Carbide Metals Company Fellow D.B. Lemmon, B.S., M.S., Ford Foundation Fellow

Work concerned with the high-temperature oxidation of cobalt-base alloys has been continued. Reproducibility of results having to do with the kinetics of high-temperature oxidation has been enhanced by the development of improved surface preparation methods. After building the appropriate apparatus, electropolishing procedures have been developed and improved. At all temperatures investigated to date, pure cobalt has followed the parabolic rate law almost ideally, and expected deviations were found only during the initial stages of oxidation. The existence of a double oxide layer, as reported by several investigators but as not found by others, has been confirmed, and X-ray diffraction procedures have been utilized to identify the various compositions involved. It has been definitely established that at temperatures in the vicinity of the Curie point there is anomalous behavior of oxidation characteristics.

Creep and stress-rupture testing of cobalt-base alloys was considerably slowed by the instrumentation and furnace failure of a number of the older testing rigs obtained on loan from an industrial research laboratory. Appropriate repairs have been made, and the several programs have been continued.

Very high-temperature, very short-time creep-rupture testing of 347 stainless steel, accomplished in a special test rig, has resulted in data that may be correlated and extrapolated, with an unusual degree of precision, by the three most common parameter methods. Correlation with two more complicated and involved parameters is being carried-out at this time. Several prospective publications dealing with the mentioned data, and also considering raw test results obtained by other workers, are now being written.

The initial doctoral thesis having to do with the transient creep of ultra-pure face-centered cubic metals is nearing completion----the thesis will be presented in late May. Materials considered include copper, aluminum, and silver. The existence of logarithmic and Andrade type creep has been verified; the transition from one type to the other, as a function of temperature and/or stress has received great attention, as have recovery effects.

Low Temperature Physics

470.19

Senior Investigator:

Research Staff:

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G.I. Kaufman, B.S.
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A. <u>Ultrasonic Attenuation in Superconductors</u>

The ultrasonic attenuation measurements are intended to determine the gap in the electronic energy levels in superconductors, in particular the anisotropy of this gap. Our present measurements in lead are being strongly affected by the dependence of the measured attenuation upon the amplitude of the ultrasonic pulse. This phenomenon, previously unobserved, is now being carefully investigated by observing its temperature, orientation, and superconducting state dependence. The energy gap studies are also being continued by using the lowest possible pulse amplitude where the results appear to be in reasonably good agreement with gap determinations by other techniques. Similar studies in superconducting mercury are now under development.

B. <u>Superconductivity and Lattice Defects</u>

Apparatus has been developed which permits electrical resistivity measurements upon specimens deformed at $4^{\rm O}{\rm K}$ and following subsequent anneal. The most interesting findings to date have been in several alloys of indium in lead. In general, as the magnetic field applied to these specimens is raised, there is a rather broad range of field in which normal resistance is gradually restored. When the specimens have been sufficiently cold worked, a sharp minimum shows up in the gradual rise in resistance. This minimum is closely associated with the ${\rm H_{c2}}$ field of type II superconductors and the "peak effect" observed in the critical current of transition element alloys. Studies of the strain and annealing dependence of this effect are being carried out to determine, if possible, the responsible defect configuration.

Ceramics Research

470.20

Senior Investigators:

R.C. DeVries, Ph.D. Professor of Ceramics J.D. Mackenzie, Ph.D. Professor of Material Science

Research Staff:

W.N. Lawless, B.Met.E. Graduate Assistant R. Haskell, B.Met.E. NDRA Fellow M. Schwartz, Graduate Assistant

A. Ceramics

One of the prime objectives of the ceramics program is the relationship of microstructure to properties in ceramic systems. In addition, we are interested in the thermodynamics and kinetics of reactions in these systems.

Current Research

As a result of the program aimed at understanding the structure of the 180° domain wall in BaTiO₃ and the nature of the polarization change across the wall two reports are being prepared. One report will be concerned with the temperature dependence of the refractive index of tetragonal BaTiO₃ by a modified Chaulnes' method; and the second with anisotropic oxygen polarizability in the carbonate minerals. In the former an accurate value of the ordinary ray index (2.368) at room temperature will be reported. In the latter report the validity of the point-dipole calculation of optical behavior will be established. Both of these studies were for the purpose of obtaining good oxygen polarizability numbers, which are critical input data for the overall computer calculation on the 180° wall.

The thermodynamic calculation from P-T data for kyanite decomposition to corundum plus an alumino-silicate liquid has been established. These results proved to be in very good agreement with recent measurements made by the Bureau of Mines.

Future Research

Besides continuation of the $BaTiO_3$ 180° domain wall treatment, we are beginning a study of deformation (mechanical) twinning on single crystal $BaTiO_3$.

B. Glass and Non-Metallic Materials

The objectives of this work are twofold: (a) to correlate properties and structures of glass and other non-metallic solids, and (b) to prepare new materials and study their properties. During this period, a new laboratory has been completed and equipment such as furnaces and controllers have been installed.

Mr. Allen Dix, graduate Assistant, has studied the crystallization behavior of BeF₂. A micro-furnace is being built so that rate measurements can be made under a microscope in a controlled atmosphere. A high temperature furnace for use up to 1800°C has been constructed. Initial attempts have been made to melt glasses in the system CaO.SiO₂ for diffusion and electrical conductivity measurements. Dr. J.D. Mackenzie, Professor of Materials Science, has been engaged on a theoretical study of semiconduction in oxide glasses. A technical report on this topic was written during this period.

Charge-Transfer Complexes

470.21

Senior Investigator:

R.L. Strong, Ph.D. Professor of Physical Chemistry

Research Staff:

F.T. Lang, B.S. Graduate Assistant

The thermodynamic properties of complexes of iodine with benzene and diethyl ether have been studied spectrophotometrically in the gas phase from 50° to 100° C. Charge-transfer absorption maxima are at 268 and 234 m, respectively, for the benzene-iodine and ether-iodine systems. These results, when compared with those for the corresponding liquid-phase complexes, suggest an appreciable solvent interaction with the complex in the liquid phase. This interaction manifests itself also in the complex formation constants, K_{298} , which are 4.5 and 6.5 liters/mole (as compared to 0.15 and 0.69 liter/mole measured in the liquid phase), respectively, for the benzene-iodine and ether-iodine complexes, and leads to a low Henry's law constant of ca. 10^{-5} liter atm./mole for the two complexes.

Attempts to extend these spectrophotometric studies to other ethers and to the aliphatic amines were unsuccessful due to rapid irreversible reaction between the components.

Dispersion-Strengthened Materials

470.23

Senior Investigator:

F.V. Lenel, Professor of Metallurgical Engineering

Research Staff:

G. Leverant, Research
Fellow
J. Hirschhorn, Research
Fellow
A. Hingorany, Graduate
Assistant
R. Oswald, Graduate
Assistant

The study of the strengthening mechanism in internally oxidized silver alloys is being continued. A paper "Internal Oxidation at Constant Velocity of Silver-Magnesium Alloys" by R.A. Bosch, F.V. Lenel, and G.S. Ansell has been submitted for publication. The investigation of internally oxidized silver-magnesium alloys has been extended to alloys oxidized at lower temperatures, i.e. 600, 700, and 800 C and a study of such alloys with lower magnesium content (under 1 atomic percent) is planned. At the same time the properties of silver-aluminum alloys after internal oxidation at oxygen pressures of several atmospheres are being measured. It is hoped also, that these studies will lead to a broadening of our understanding of the strengthening mechanism in the internally oxidized silver alloys.

Measurements of the internal friction spectrum of internally oxidized silver-magnesium alloys are under way and characteristic differences between the spectra of pure silver, of a silver-magnesium alloy and internally oxidized silver-magnesium alloy have been found. An attempt will be made to interpret these differences. An apparatus for measuring the creep of internally oxidized silver-magnesium alloys has been set up.

The study of the structure of internally oxidized columbiumzirconium alloys both by electron and by optical micrography and the correlation of the observed structure with the hardness and the lattice parameter of the alloys is nearing completion.

Corrosion Studies

470.25

Senior Investigator:

N.D. Greene, Associate Professor of Metallurgical Engineering

Research Staff:

H. Cleary, Graduate Assistant

Funds from this grant were used to partially support basic research studies of the palladium hydrogen diffusion electrode which have been completed.

This electrode has unique properties, including high power output at ambient temperatures, which make it a promising electrode for fuel cells. Studies have included measurement of the diffusion coefficient of hydrogen (protons) through palladium membranes, the effect of surface catalysts on proton flux, and critical experiments to determine the controlling step during diffusion and subsequent electrochemical oxidation of hydrogen.

Future plans include a study of metal dissolution kinetics at very high applied currents, completely supported by grant funds. Understanding the behavior of metals and alloys under these conditions is important in such fields as electropolishing, electrodeposition, and electrochemical machining.

Nuclear Magnetic Resonance Research

470.26

Senior Investigator:

P.A. Casabella, Ph.D., Assistant Professor of Physics

Research Staff:

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T. Oja, B.S.
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D.D. Peterson, B.S.
Graduate Assistant
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Oak Ridge Institute
of Nuclear Studies Fellow

The nuclear quadrupole coupling constant of Na²³ in Rochelle salt has been determined by studying the splitting of the nuclear magnetic resonance in a single crystal. Complete rotation patterns were obtained at 28°C, in the high temperature paraelectric phase, and at 3°C in the ferroelectric phase. The crystal was then fixed at one orientation, and the resonance was studied as the temperature was raised from 3°C to 28°C. Over a small temperature range about

the curie temperature of 24°C, the resonance pattern was observed to change continuously from the low temperature to the high temperature pattern. The continuous nature of the change confirms that the ferroelectric transition is of second order. However, the fact that the resonance is observable, and unbroadened throughout the transition is in disagreement with some existing local field theories that have been advanced to explain the ferroelectricity of Rochelle Salt. The investigation of this matter is being continued.

X-Ray Scattering

470.28

Senior Investigator:

J.L. Katz, Ph.D. Associate Professor of Physics

Research Staff:

R.H. Wilson, Graduate Assistant E.F. Skelton, Graduate Assistant K. Dunn Undergraduate Assistant

The temperature dependence of the Debye-Waller factor has been measured for three iron-nickel alloys, 29, 31 and 33 atomic percent nickel, through the curie temperature. X-ray Debye temperatures based on the Debye-Waller factors have been computed and are being compared with the temperature dependence of the Debye temperatures calculated from the elastic constant data of Alers, Neighbors and Sato (J. Phys. and Chem of Solids 13, 40 (1960)). Theoretical expressions for correcting the X-ray intensity measurements for thermal diffuse scattering have been developed. The curie temperatures of the three compositions have been measured. These measurements support the X-ray experiments. In addition experimental determination of the Debye-Waller factor for pure nickel is in progress.

This work is being continued with several new compositions at temperatures down to liquid helium temperatures.

Other studies including real time computer operation of X-ray instrumentation and the investigation of the Group IIIb metal-trihalides is in progress.

TECHNICAL PAPERS

Abrahams, A., Wiberley, S.E., Nachod, F.C.
"Proton Magnetic Resonance of Some Cyclopropane Derivatives"
Submitted to <u>Journal of Applied Spectroscopy</u>

Casabella, P.A., Miller, N.C.
"A127 Quadrupole Coupling in Solid A1C13"
Submitted to <u>Journal of Chemical Physics</u>

Love, R.E., Shaw, R.W.
"Ultrasonic Attenuation in Superconducting Lead"
Submitted to Reviews of Modern Physics

Hirschhorn, J.S.

"Determination of Solute Diffusivity from Internal
Oxidation Experiments"

Submitted to <u>Transactions of the Metallurgical</u>
Society of AIME for publication as a technical note

Bosch, R.A., Lenel, F.V., Ansell, G.S.
"Internal Oxidation at Constant Velocity of SilverMagnesium Alloys"
Submitted to <u>Transactions of the Metallurgical</u>
Society of AIME

Mackenzie, J.D.

"Semiconducting Oxide Glasses"

To be published as a chapter in a book entitled

Modern Aspects of the Vitereous State; Butterworth's
Publishing 1964

APPENDIX A

Members of Interdisciplinary Materials Research Center Faculty Committee

S.E. Wiberley, Chairman	Professor of Analytical Chemistry
E.B. Allen	Professor of Mathematics and Astronomy and Dean of the Graduate School
G.S. Ansell	Associate Professor of Metallurgical Engineering
W.H. Bauer	Professor of Physical Chemistry, Dean of the School of Science
A.A. Burr	Professor of Metallurgical Engineering, Dean of the School of Engineering
R.H. Hartigan	Director of the Research Division
H.B. Huntington	Professor of Physics, Chairman of the Department of Physics
G.J. Janz	Professor of Physical Chemistry
F.V. Lenel	Professor of Metallurgical Engineering
J.M. LoGiudice	Administrative Director of the Interdisciplinary Materials Research Center